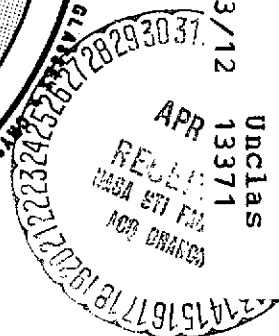
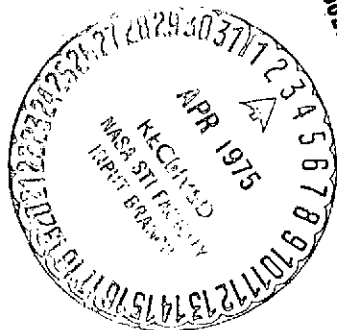


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INTRODUCTION TO SPACE PROCESSING

By

Mathias P. L. Siebel
Process Engineering Laboratory
Marshall Space Flight Center
Alabama 35812

ABSTRACT

Activities in space processing which have culminated in the Skylab experiments are described. Particular emphasis is given to the zero-g experiments and demonstrations performed in earth laboratories; in the MSFC drop tower; and on Apollo flights. It is shown that both the experiments and the processing apparatus have evolved and developed through the experience gained on successive low-g tests.

The status of the activities related to space processed materials such as crystals, composites, eutectics, immiscible compounds, glasses and biologicals is discussed. Examples are taken to show that some materials, such as crystals and immiscible intermetallic compounds have developed through several phases while others such as glasses and biologicals are still in the early stages of development.

Studies and tests have been performed to gain understanding of the physics of the low-g processes; in this connection the electrophoresis and heat flow and convection demonstrations flown on Apollo missions are briefly described. Knowledge on heating, cooling, diffusion, surface tension and solidification effects in low-g has been provided by the analysis of various experiments.

The development of processing equipment has led to apparatus for containerless processing, to various furnaces and to other devices for heating, cooling and containing materials. The paper concludes with a description of the Skylab M512 Materials Processing Facility.

SKYLAB EXPERIMENT M552
EXOTHERMIC BRAZING

By

Clyde M. Adams, Jr.
University of Wisconsin
Milwaukee, Wisconsin 53211

ABSTRACT

Specimens composed of pure nickel and of stainless steel were used to explore the effects of space environment on capillary flow and metallurgical reaction during exothermic brazing with silver-copper-lithium alloy as the filler metal. In the absence of gravity, only surface tension forces are operative in capillary action, and fully developed flow is realized in much wider gaps than those considered practical for terrestrial brazing.

The absence of gravity greatly enhances the prospects of brazing as a joining process in space.

Certain solution reactions, such as the dissolving of nickel in a silver-rich liquid, are substantially advanced in the zero gravity environment, although solidification structures, such as the formation of the silver-copper eutectic, appears to be gravity insensitive.

SKYLAB EXPERIMENT M551
METALS MELTING

By

E. C. McKannan, R. M. Poorman
Astronautics Laboratory
Marshall Space Flight Center
Alabama 35812

ABSTRACT

The objectives of the M551 Metals Melting Experiment are to (a) study behavior of molten metal, (b) characterize metals melted and solidified in the low gravity space environment compared to one-gravity of earth, and (c) determine feasibility of joining metals in space. The experiment used the electron beam (EB) and chamber of the M512 apparatus to make a dwell puddle and a weld in a rotating disc of varying thickness. Hence, the EB performed cut-through, full and partial penetration welds, in addition to a re-melt button. The three disc materials were aluminum 2219-T87, 304 stainless steel and pure tantalum to provide a wide range of density and melting conditions. Observations to date include (a) the proof that EB welding, cutting and melting can be done successfully in low gravity. Earlier, some welding authorities had postulated that without gravity the EB would force the molten puddle out of contact. However, the experiment proved that surface tension forces predominate. (b) From the view-point of cast-solidification, small, equiaxed grains in Skylab specimens compared to large, elongated grains in ground-based specimens were observed. The former are thought to be associated with constitutional super-cooling and nucleation where the latter are associated with dendritic solidification. In further support of the more equiaxed grain growth in Skylab, symmetric subgrain patterns were frequently observed where there was much less symmetry in ground-based specimens. Further work is being done to explain the differences in solidification.

SKYLAB EXPERIMENT M553
SPHERE FORMING EXPERIMENT

By

D. J. Larson, Jr.
Grumman Aerospace Corp.
Bethpage, New York 11714

ABSTRACT

The M553 Sphere Forming Experiment was conducted in the M512 Furnace Facility during Skylab II. The experiment was designed to study the effects of weightlessness on solidification processes. Four face centered cubic materials of varying alloy contents and solidification reaction types were studied.

Careful analysis of the samples indicated that there was an outstanding record of both initial and terminal solute redistribution processes and that this record may be substantially better than that obtained terrestrially for comparable experimental conditions. Further, the last regions to solidify evidenced extensive solidification terracing, and for one alloy type this terracing was found to be decorated with second phase precipitate particles. These particles were highly localized in systematic arrays which were frequently low index crystallographic systems.

Some of the samples underwent unanticipated solidification reactions that involved solid/liquid/gas reactions rather than the anticipated solid/liquid processes. Subsequent consideration indicated that this phenomenon could have been anticipated and that it was a result of the reduced gravity environment - that is, the reduced gravity environment magnified a typically microscopic phenomenon (terrestrially) so that it became a macroscopic effect. Work is continuing on quantifying this effect.

It is felt that these and subsequent results will contribute to the detailed understanding of terrestrial solidification processes and have shown the importance of considering the gas phase during space processing. Whereas prior consideration of one-g solidification has previously dealt with liquid/solid reactions, this work indicates that consideration of solid/liquid/gas reactions are important in reduced gravity and are potentially beneficial.

SKYLAB EXPERIMENT M479
ZERO GRAVITY FLAMMABILITY

By

J. H. Kimzey
Lyndon B. Johnson Space Center
Houston, Texas 77058

ABSTRACT

Flammability under conditions of weightlessness was investigated on Skylab 4. Thirty-seven tests using six materials were successfully carried out to learn more about how a flame acts in the absence of convection. Specific objectives were to note the extent of surface flame propagation and flash-over to adjacent materials, rates of surface and bulk flame propagation, self-extinguishment, and extinguishment by both vacuum and water spray. Data were returned in the form of crew comments on voice tapes and sixteen millimeter motion pictures taken at 24 frames per second. All tests were performed and all hardware functioned properly, although an operational oversight caused the water in the supply tank to be less than the minimum required pressure. Burning rates were significantly reduced as anticipated from earlier aircraft tests. Some reduction of total burning was also noted. The surface burn was not followed by continued inward burning as typically experienced on earth although some one-g tests at extremely low pressures have partially reproduced this condition. Ignition and extinguishment also appear to be similar to one-g. The typical soft-blue flame was seen by the crew, but was not detected on the film emulsions used. Smoke patterns were also noted. This can be of value in studying Brownian movement of solids in gases under conditions of weightlessness.

SKYLAB EXPERIMENT M518
MULTIPURPOSE FURNACE

By

W. R. Adams
Skylab Program Office
Marshall Space Flight Center
Alabama 35812

ABSTRACT

The decision to perform materials processing experiments as part of the basic Skylab mission led to the development of a multipurpose electric furnace system capable of useful operation up to 1000° centigrade. The furnace facility, in which eleven individual experiments were performed, met all design requirements by automatically processing each experiment after a crew member had "dialed in" an appropriate temperature level, period of time for which the furnace would hold the desired temperature, and specific cool down rate desired by each Principal Investigator.

The furnace system, designated M518, was operated during the second and third Skylab missions to process eighteen sets of experiment samples. This effort included the processing of prime sample ampoules for all eleven selected experiments, and an additional "run" utilizing the secondary set of samples from seven of the eleven at slightly different parameters.

The apparent success of the entire M518 Skylab operation had led to the production of a similar experiment package for the Apollo-Soyuz mission planned for mid-1975.

SKYLAB EXPERIMENT M557
IMMISCIBLE ALLOY COMPOSITIONS

By

J. L. Reger
TRW Systems Group
Redondo Beach, California 95560

ABSTRACT

Ampoules containing materials exhibiting either liquid or solid state immiscibility were thermally processed in the M-518 Multipurpose Electric Furnace under low and one gravity conditions. Three cartridges, each containing three separate experiment ampoules were processed simultaneously during one furnace run.

Two of the experiment ampoules, gold-germanium (Ampoule A) and lead-zinc-antimony (Ampoule B), were processed in the isothermal portion of the furnace and one experimental ampoule, lead-tin-indium (Ampoule C), was processed in the gradient region of the furnace. Both the control (one gravity) and low gravity processed specimens were analyzed for their metallographic and electronic properties.

In all cases, the low gravity processed specimens exhibited better homogenization and microstructural appearances than the one gravity control specimens. The electronic behavior of the low gravity specimens were equal or superior in every respect and the ampoule B specimens exhibited an anomalous superconducting transition temperature approximately 2 K higher than either the elements or the one gravity control specimens. In addition, the low gravity processed Ampoules A and B exhibited X-ray diffraction lines not identifiable with any referenced diffraction patterns.

From these analyses, it is concluded that low gravity processing of materials processing liquid or solid immiscibility can produce compositions exhibiting unusual metallographic and electronic behavior.

SKYLAB EXPERIMENT M565
SILVER GRIDS MELTED IN SPACE

By

A. Deruyttere, E. Aernoudt, H. Goeminne, J. Smeesters
O. Arkens, M. Verhaeghen
Department Metaalkunde, Universiteit te Leuven
Leuven, Belgium

ABSTRACT

The objective of the experiment was to make a preliminary study of the behaviour of porous material when melted and resolidified in weightless condition.

Two kinds of samples have been used: perforated discs and a fibre compact. The observations made on the various specimens are presented and compared with those made on similar specimens treated in the same manner on the ground.

Some provisional conclusions are:

- (1) most of the original porosity in the samples has disappeared during the melting stage. This could possibly be avoided by different heating and pressure conditions or by using samples with closed pores. If necessary, the mobility of the pores could probably be reduced by the presence of a slowly diffusing gas.
- (2) even if samples were perfectly spherical in the liquid state, their shape is altered on solidification due to both shrinkage pipe formation and constitutional supercooling.
- (3) the leveling out of impurity concentration gradients appears indeed to be slow in the molten metal when gravity induced convection is absent.
- (4) when only a part of a solid body is melted in zero gravity, the tendency of the molten part to become spherical may be much restricted.

SKYLAB EXPERIMENT M561
WHISKER REINFORCED COMPOSITES

By

T. Kawada, S. Yoshida, Y. Yoda, S. Takahashi, E. Ozawa
National Research Institute for Metals
Tokyo, Japan

ABSTRACT

The object of the experiment is to get Ag and SiC whisker composites with high density and uniform distribution of whiskers by heating and pressurizing semisintered products above the melting temperature of Ag under a weightless environment.

This report describes the results of examination carried out on the samples processed on Skylab and comparison with those prepared on the ground. The nominal volume fraction of SiC is 2, 5, and 10%. The size of each sample is 8mm in diameter and 30mm in length. The microhardness is measured on a longitudinal section of the sample. Also the distribution density of whiskers is estimated by observing the section under an optical microscope.

The result of hardness measurement shows that the scatter of hardness value in the Skylab samples is generally small as compared with the ground based samples. Macroscopic segregation of whiskers toward the upper surface is clearly observed for the ground based samples. Such segregation is not observed for the Skylab samples.

SKYLAB EXPERIMENT M556
VAPOR GROWTH OF IV-VI COMPOUNDS

By

H. Wiedemeier, F. C. Klaessig
Rensselaer Polytechnic Institute
Troy, New York 12181

ABSTRACT

The analysis of GeSe and GeTe single crystals grown by vapor transport on earth and in micro-gravity environment yielded results of scientific and technological significance.

Under ground-based conditions, mass transport rate studies of GeSe and GeTe as a function of transport agent (GeI_4) pressure revealed the existence of three different transport regions, in which the mass transport is controlled by the rate of the heterogeneous solid-gas phase reaction, by gas phase diffusion and by gravity-driven convection, respectively. These studies demonstrated a decrease in crystal quality with increasing contribution of the convective component to the overall transport process. Thus, the degree of perfection of earth grown crystals is limited by the negative effects of convection.

The experimental conditions selected for the Skylab studies corresponded to a low (GeSe), medium (GeTe) and high (GeSe) convective contribution under ground-based conditions. A comparison of earth and space crystals demonstrates the improved quality of crystals obtained in micro-gravity environment in terms of growth habit, surface and bulk morphology. The significant observation of considerably greater material fluxes in space than expected is of technological importance for the growth of crystals of higher quality and at reasonable rates in the absence of gravity. The experimental evidence for the existence of transport modes not considered in present vapor transport models is of basic scientific value. These findings demonstrate the unique conditions of micro-gravity for crystal growth and for observing fundamental facts of transport and condensation phenomena.

SKYLAB EXPERIMENT M560
GROWTH OF SPHERICAL CRYSTALS

By

H. U. Walter
Department of Physics
The University of Alabama in Huntsville
Huntsville, Alabama 35807

ABSTRACT

Directional solidification of a containerless melt that was suspended at the end of a seed crystal was employed to produce single crystals of InSb during Skylab missions. Extremely well developed growth facets ((110) and (111)) are observed. With undoped crystals, facets and sub-facets are flat within a few hundred \AA . Material produced under steady state growth conditions shows continuous improvement of structural perfection; typically dislocation densities, as revealed by etching, are reduced by a factor of 5 to 10 over a distance of 1 cm to 20 - 30 dislocations per cm^2 . Incorporation of dopant (selenium) is homogeneous during steady state growth.

Based on this investigation, the following conclusions concerning processing of single crystals from the melt in low gravity environment can be reached: Steady state solidification can be achieved to an extent that is not possible on earth. As a result, chemically homogeneous single crystals with high structural perfection can be prepared. As shown with this study, containerless processing of single crystals in space is suitable in general; it should prove especially valid for contamination-free processing of highly reactive and high melting temperature materials.

SKYLAB EXPERIMENT M562
INDIUM ANTIMONIDE CRYSTALS

By

A. F. Witt, H. C. Gatos, M. C. Lavine,
M. Lichtensteiger & C. J. Herman
Massachusetts Institute of Technology
Cambridge, Massachusetts 02139

ABSTRACT

Crystal growth and segregation under zero gravity conditions were studied by melting and resolidifying in space portions of InSb crystals grown on earth. Morphological features of tellurium-doped indium antimonide grown in space manifest virtually unconfined solidification whereby neither the melt nor the crystal were in intimate contact with the confining wall during growth. Dynamic surface tension effects, presumed to be a potential interference to effective materials processing in space, were found to remain localized on the surface and to not affect growth and segregation in the bulk. It was found that compositional fluctuations originating from gravity-induced convection in earth-grown crystals are absent in the space-grown material. Segregation behavior associated with peripheral facet growth was directly related to nucleation effects which could not be identified in earth-grown crystals because of convective interference. The analysis of rotational twin bands present in the space-grown material led to the unambiguous confirmation of the theory of rotational twin formation. The present results demonstrate the uniqueness of outer space for processing materials and for establishing fundamental facts of solidification.

SKYLAB EXPERIMENT M563
MIXED III-V CRYSTAL GROWTH

By

W. R. Wilcox, J. F. Yee, Mu-Ching Lin,
Kalluri Sarma and Sanghamitra Sen
University of Southern California
Los Angeles, California 90007

ABSTRACT

InSb and GaSb form a continuous series of solid solutions with a variety of important applications if high quality single crystals can be routinely produced. Directional freeze experiments were performed on earth and in Skylab in order to determine the effects of gravity on crystallization of such alloys. The melts in the first set of Skylab samples apparently did not contact the crucible wall during solidification, while it did in the second set run at a higher soak temperature. All samples were polycrystalline with grains in the slow regrowth region elongated in the growth direction. The grain size was about the same in all cases. Grain boundaries were much more difficult to distinguish in the Skylab samples. The Skylab samples had many fewer twins, but a few more microcracks. Preliminary experiments on the ground with a transverse magnetic field available during solidification showed less twinning when the field was imposed. Gas bubbles were concentrated near the initial solid-liquid interface in the earth samples, and much more uniformly dispersed in the Skylab samples. The solid first regrown was homogeneous within detection limits, but eventually became inhomogeneous. This happened sooner with the Skylab samples and with the vertically solidified one 'g' samples than with the horizontally solidified one 'g' samples.

SKYLAB EXPERIMENT M559
MICROSEGREGATION IN GERMANIUM

By

J. T. Yue, F. W. Voltmer
Semiconductor Research and Development Laboratory
Texas Instruments, Incorporated
Dallas, Texas 75222

ABSTRACT

The results of Skylab Experiment M559 on the influence of gravity-free solidification on microsegregation are reported. Two identical sets of 111 oriental germanium crystals were prepared, melted, and resolidified with the gradient freeze technique by normal freezing in Westinghouse furnaces. One set was melted and resolidified in space aboard Skylab Mission No. 2, the other set on earth. Each set of three germanium crystals were doped with gallium, antimony, and boron initially with respective concentrations of 7.8×10^{16} atms/cc., 0.42×10^{15} atms/cc., and 2×10^{15} atms/cc. Detailed spreading resistance measurements on both the space grown and terrestrially grown crystals indicate an improvement in solute microsegregation during space resolidification. The improvement is attributed to the absence of gravity induced convective mixing. The effective segregation coefficients appear to be higher for solidification in space, indicating a thicker solute boundary layer at the solid-liquid interface. The improvement in solute microsegregation in space is accompanied with a solid-liquid interface structure which ranges from planar to concave toward the melt during crystal growth. These interface shapes are contrasted with the convex type toward the melt encountered during terrestrial crystal growth. A theoretical model is presented which explains the observed microsegregation behavior in a gravity-free environment.

SKYLAB EXPERIMENT M558
RADIOACTIVE TRACER DIFFUSION

By

A. O. Ukanwa
Howard University
Washington, D. C. 20001

ABSTRACT

The objective of the M558 Radioactive Tracer Diffusion experiment was to measure self-diffusion and impurity diffusion effects in liquid metals in space flight and characterize the disturbing effects, if any, due to spacecraft acceleration. Because of the near-zero gravity aboard Skylab, the effects of convection on pure self-diffusion would be minimal.

Three cartridges were tested in Skylab. Each cartridge encapsulated a neutral zinc rod carrying a section of radioactive zinc-65. The radioactive sections were at the thermally cold end of one zinc-rod, at the thermally hot end of another zinc-rod and at the central section of the third zinc rod. The diffusion time involved 2 hours of heat-up, 1 hour of soak under a temperature gradient and 13 hours of passive cool down in the M518 Multipurpose Electric Furnace. Three cartridges, configured similar to the Skylab cartridges, had also been tested on earth to provide ground-base data under normal gravity for comparison.

Data from the Skylab samples have been markedly different from the ground-base data obtained on earth. On earth, radioactive zinc-65, propelled by gravity-induced convection, rapidly diffused through the neutral zinc samples in less than 1 hour of soak time, to yield an almost uniform distribution of zinc-65 throughout the sample. On the other hand, in the near-zero gravity environment of Skylab, the slow distribution of zinc-65 revealed the pattern attributable mostly to pure volume diffusion in the absence of appreciable convective currents. Thus coefficient of self-diffusion for liquid zinc, not warped by convective effects, was calculated. The activation energy for self-diffusion which was determined was not significantly influenced by gravity.

SKYLAB EXPERIMENT M566
COPPER-ALUMINUM EUTECTIC

By

Earl A. Hasemeyer
Process Engineering Laboratory
Marshall Space Flight Center
Alabama 35812

Charles V. Lovoy
Astronautics Laboratory
Marshall Space Flight Center
Alabama 35812
and

L. L. Lacy
Space Sciences Laboratory
Marshall Space Flight Center
Alabama 35812

ABSTRACT

The purpose of the experiment is to evaluate the effects of reduced gravity in the orbiting Skylab vehicle on termination faults and other defects in aligned eutectic material. Specimens of copper-aluminum eutectic (67% wgt. aluminum - 33% wgt. copper) were partially melted in the M518 Multipurpose Electric Furnace then directionally re-solidified. The six specimens processed in Skylab 3 and 4 are being characterized by scientific laboratories. Changes in physical properties, quantitative metallography, and chemical homogeneity are being investigated, and will be reported.

SKYLAB EXPERIMENT M564
METAL AND HALIDE EUTECTIC

By

A. S. Yue, J. G. Yu
University of California
Los Angeles, California 90024

ABSTRACT

Fiberlike NaCl-NaF eutectic mixtures have been produced on earth and in space by the directional solidification technique. It was found that continuous and discontinuous NaF fibers were embedded in a NaCl matrix from ingots grown in space and on earth, respectively. The production of continuous fibers in a eutectic mixture was attributed to the absence of convection current in the liquid during solidification, in agreement with predictions.

Macroscopic and microscopic examinations on longitudinal and transverse sections of space - grown and earth - grown ingots were made. It was found that during the major portion of the space solidification process, the NaF fibers were aligned with the ingot axis. However, they were normal to it during the very beginning of the solidification process. This indicated that the direction of heat flow was perpendicular to the ingot axis. The best microstructures were obtained from ingots grown in space. These microstructures were compared with those produced on earth with and without convection current in the liquid during growth.

Optical transmittance measurements of transverse and longitudinal sections of the space - grown and earth - grown ingots were carried out with a polarizer in a Perkin Elmer Spectrometer. It was found that for a given sample thickness, the highest percentage of transmittance was obtained from ingots grown in space. The effect of sample thickness on transmittance was investigated. It was found that the thinner the sample, the higher the transmittance over a range of wavelengths, in agreement with the general optical property of transparent materials exposed to electromagnetic waves.

SKYLAB SCIENCE DEMONSTRATIONS

By

T. C. Bannister
Space Science Laboratory
Marshall Space Flight Center
Alabama 35812

ABSTRACT

Twelve science demonstrations were accomplished on Skylab III and IV. These science demonstrations were defined in response to crew requests for time gap-fillers and were designed to be accomplished using on-board equipment. Nine of these science demonstrations were in the area of materials science and space processing. The nine science demonstrations will be described and preliminary results given. A short film on fluid mechanics in orbit will be shown. The nine science demonstrations include the following: Diffusion in Liquids, Ice Melting, Liquid Floating Zone, Immiscible Liquids, Liquid Films, Rochelle Salt Growth, Deposition of Silver Crystals, Fluid Mechanics Series, and Charged Particle Mobility.

SPACE PROCESSING ACHIEVEMENTS

By

Ernst Stuhlinger
Associate Director for Science
Marshall Space Flight Center
Alabama 35812

ABSTRACT

Skylab offered an unprecedented opportunity for a variety of experiments related to materials processing and manufacturing under weightlessness. A number of different observations were planned and carried out, and all were successful as experiments. Some of them surpassed all expectations. As a result, our basic knowledge of materials processes, and particularly of the role of gravity in these processes, has made substantial progress. Besides increasing our basic knowledge, these experiments have provided direct evidence and proof that a number of important processes work very well under zero gravity, among them the cutting and welding of metals with an electron beam; the joining of metals by brazing with exothermic heat sources; the manufacturing of large single crystals of very high purity and homogeneity either by recrystallization from melts, or by vapor deposition growth; the homogeneous distribution of dopants in semiconductors; the diffusion of liquids without convective currents; the alloying and even chemical compounding of metals which do not mix in earth-produced samples; the homogeneous distribution of components in composite materials; and the production of new superconductors. These results were achieved in Skylab experiments although the experimental parameters had not yet been optimized. Even from this first set of space experiments, it has become obvious that gravity is indeed a potent factor in the limitation of purity, homogeneity, and perfectness of earth-produced materials. The Skylab experiments are pointing toward a very promising and elaborate program of zero-gravity technology on the Shuttle, and beyond.

SPACE PROCESSING PAYLOADS FOR SPACELAB

By

Kenneth R. Taylor
Program Development
Marshall Space Flight Center
Alabama 35812

ABSTRACT

This paper will address the results and goals of the space processing payload definition activities. These activities encompass the definition of design envelopes for payload equipment items and the system engineering required to integrate these items into various payloads, which can serve the research needs of space processing Principal Investigators.

Concepts which permit the flying of partial or dedicated payloads will be presented. These concepts are based on the modular (building block) approach to payload equipment design and integration.

SOUNDING ROCKETS FOR SPACE PROCESSING APPLICATION EXPERIMENTS

By

Brian O. Montgomery
Science Engineering Directorate
Marshall Space Flight Center
Alabama 35812

ABSTRACT

This paper is to outline and discuss a program being considered by NASA for conducting scientific experiments in Space Processing Applications by use of Sounding Rockets on a routine, repetitive basis.

During the coast phase of the ballistic trajectory of a Sounding Rocket, above 90Km, a suitably stable low level of gravity ($10^{-3}g$ or less) is available for 415 seconds for a 100 pound net experiment payload weight, or 350 seconds for a net experiment payload weight of 250 pounds.

The physical and dynamic characteristics of the Aerobee 200 sounding rocket are discussed. Payloads are recoverable for post flight analysis.

The basic processing and measuring equipment required to conduct experiments and verify the environment are discussed. Equipment is to be refurbished after each flight and reflown.

The protocol for participation in the program by scientists outside of NASA is explained.

The current status of the proposed program is reviewed.

FURNACE SYSTEMS DEVELOPMENT

By

B. R. Aldrich
Process Engineering Laboratory
Marshall Space Flight Center
Alabama 35812

ABSTRACT

A review of MSFC's past space flight furnace development, current furnace systems now under development for the Apollo-Soyuz and Sounding Rocket programs, and a look into the future, at advanced furnace systems including high temperature capabilities, containerless melting and automated processing systems will be presented.

Successful scientific experiments in the field of materials processing have been conducted on Apollo flights and Skylab missions. Materials processing experiments are also planned for the Apollo-Soyuz mission in 1975 and for the Shuttle/Spacelab program beginning in the latter part of this decade. Additionally, many basic studies and scientific experiments will be performed in preparation for the next long duration manned mission, at relatively low cost, through use of short duration flights available with sounding rockets.

Materials processing in space has originated the need to develop new and unusual processing facilities. The experimenter's requirements for furnace systems are continually increasing in complexity and sophistication. In order to provide the desired processing parameters, the capabilities of space flight experiment hardware must keep pace with the increasing scientific needs.

SYSTEM DESIGN FOR FREE FALL MATERIALS PROCESSING

By

R. G. Seidensticker
Westinghouse Research Laboratories
Beulah Road, Churchill Borough
Pittsburgh, PA 15235

ABSTRACT

The boundry conditions for free fall materials processing systems are generated by the requirements of the flight vehicle and not by the absence of gravitational acceleration itself. The absence of gravity can, in fact, greatly simplify the analysis of a processing system. In order to take advantage of this situation, however, the experiment requirements must be carefully defined in terms of the required physical condition rather than the more usual case of apparatus operating parameters. The design of the materials processing system then becomes an exercise in achieving the necessary thermal and mechanical state of the material within the operational restraint of the flight vehicle and crew.

ELECTROMAGNETIC CONTAINERLESS MELTING AND SOLIDIFICATION IN THE WEIGHTLESS ENVIRONMENT

By

R. T. Frost
General Electric Company
Space Sciences Laboratory
Valley Forge, Pennsylvania 19101

ABSTRACT

The extension of terrestrial levitation melting experiments to the weightless environment of space offers many new possibilities for basic materials science experiments and for the preparation of new materials. In the weightless environment only small position control forces are required which allows the consideration of materials whose resistivities are six orders of magnitude higher than that of the best conductors. This regime encompasses a large number of materials and containerless processes which appear to be of great interest and includes metals, immiscible alloy systems, and many interesting carbides, nitrides, borides, silicides, sulfides and beryllides. If specimen preheating is considered, some metallic oxides, semiconductors and a large number of chalcogenic glasses can also be handled. The possibility for decoupling of the positioning and heating functions in the weightless environment will allow the achievement of large undercoolings in many of these materials to explore the formation of new amorphous structures. Recent development work on appropriate techniques will be reviewed as well as examples of ground based work to explore specific candidate materials processes of greatest interest.

ACOUSTIC POSITIONING FOR CONTAINERLESS PROCESSING

By

R. R. Whymark
Intersonics, Incorporated
Chicago, Illinois 60611

ABSTRACT

An acoustic positioning system is described that is adaptable to a range of processing chambers and furnace systems. Operation at temperatures exceeding 1000°C is demonstrated in experiments involving the levitation of liquid and solid glass materials up to several ounces in weight. The system consists of a single source of sound that is beamed at a reflecting surface placed a distance away. Stable levitation is achieved at a succession of discrete energy minima contained throughout the volume between the reflector and the sound source. Several specimens can be handled at one time. Metal discs up to 3 inches in diameter can be levitated, solid spheres of dense material up to 0.75 inches diameter, and liquids can be freely suspended in 1-g in the form of near-spherical droplets up to 0.25 inch diameter, or flattened liquid discs up to 0.6 inches diameter. Larger specimens may be handled by increasing the size of the sound source or by reducing the sound frequency. Shaping of the freely suspended liquid drops is accomplished by adjusting the sound pressure. The system appears free of significant instabilities - constraining forces on the specimen are measured to be about 15% of the force needed to overcome gravity. Electrical power for terrestrial position control is about 100 to 150 watts. Chamber dimensions may be of the order of several inches to several feet. Capillary injection methods are described for introducing liquid materials into the position control system, forming critical size droplets without significant material loss. Solid specimens are injected by acoustic lift-off of the specimen from a fine wire mesh screen placed close to the desired levitation point (energy well); the wire mesh screen is substantially transparent to the sound. Powder specimens are injected by acoustic lift-off of the powder from a wire screen - the sound field agglomerates the powder into a powder ball that positions itself within the nearest energy well. Experimental results are presented for the supercooling of organic materials and for water. Benzophenone is shown to be supercooled by 10% of the melting point temperature. Estimates are presented on the increase in nucleation rate caused by the sound field. Drop tower investigations are reported for the containerless melting and solidification of chalcogenide glass.

ACOUSTIC CHAMBER FOR SPACE PROCESSING

By

T. G. Wang, M. M. Saffren, D. D. Elleman
Jet Propulsion Laboratory
California Institute of Technology
Pasadena, California 91103

ABSTRACT

Many of the processes to be carried out in Space Processing require the manipulation and control of weightless molten material. An acoustical method is being developed at JPS to form, position, and control molten material including materials that are electrically non-conducting. The program is structured in two parts: the first consists of development of a stable acoustic chamber for space processing, including an automatic feedback system, a melting and solidification system and remote control capability. The second part of the program will use the apparatus to explore the feasibility of material management both in sounding rocket flights and ultimately on the space shuttle to support the space processing program.

FLUID MOTION IN A LOW-G ENVIRONMENT

By

P. G. Grodzka

S. V. Bourgeois

M. R. Brashears

L. W. Spradley

Lockheed Missiles & Space Company, Inc.
Huntsville, Alabama 35807

ABSTRACT

The importance of natural convection and other fluid motions in low-g space processing is now well recognized. Recent space experiments in the areas of natural convection and material processing, as well as results of theoretical studies, have yielded much needed information on fluid behavior in low-g environments. The state of knowledge of fluid motions in low-g environments is reviewed and the dimensional analysis approach used to assess the relative importances of various driving forces for fluid flow in four of the Skylab material processing experiments outlined. Results of dimensional analyses for the Skylab experiments, subsequently confirmed by actual space data, are presented. Finally, the limits of dimensional analysis in assessment studies are indicated.

ROLE OF GRAVITY IN PREPARATIVE ELECTROPHORESIS

By

M. Bier, M. J. Binder, M. Coxon, J. O. N. Hinekley, T. W. Nee,
M. O. Scully, A. J. K. Smolka
Veterans Administration Hospital and University of Arizona
Tucson, Arizona 85723

ABSTRACT

Electrophoresis is a widely used technique for separation of biological macromolecules. Earthbound electrophoresis has nevertheless failed in two important respects: (a) large scale preparative separations, and (b) separation of living cells with sharp resolution. Both of these failures are at least in part attributable to effects of gravity, causing convective flows in free fluids. These arise from density differences between solutions and pure solvents (buffers) and are compounded by thermal effects arising from Joule heating. Three space experiments have already shown that these difficulties are overcome at zero gravity.

Numerous other problems remain at least partially unsolved, such as sample introduction and withdrawal of separated fractions; general concepts of optimal equipment design for space processing, control of electroosmosis; and maintenance of viability of living cells in prolonged space experimentation. These are presently objects of intensive studies here and abroad.

Solution of these problems is complicated by the fact that electrophoresis is not a single technique, but a family of related methodologies, and there is no consensus if a single manufacturing technique will answer all requirements for space processing. Our own studies center on isotachopheresis, and comprise a theoretical, engineering, and experimental analysis of possible solutions.

PREPARATIVE ELECTROPHORESIS OF LIVING LYMPHOCYTES

By

C. J. van Oss, P. E. Bigazzi, C. F. Gillman, R. E. Allen
Department of Microbiology and The Center for Immunology
School of Medicine, State University of New York at Buffalo
Buffalo, New York 14214

and

Robert S. Snyder
Astronautics Laboratory
Marshall Space Flight Center
Alabama 35812

ABSTRACT

Vertical liquid columns containing low molecular weight dextran density gradients can be used for preparative lymphocyte electrophoresis on earth, in simulation of zero gravity conditions. Another method that has been tested at one 'g' is the electrophoresis of lymphocytes in an upward direction in vertical columns. By both methods up to 10^7 lymphocytes can be separated at one time in a 30 cm glass column of 8 mm inside diameter, at 12 V/cm, in two hours. Due to convection and sedimentation problems, the separation at one 'g' is less than ideal, but it is expected that at zero gravity electrophoresis will prove to be a uniquely powerful cell separation tool. The technical feasibility of electrophoresing inert particles at zero 'g' has been proven earlier, during the flight of Apollo 16.

MATERIALS SCIENCE AND MANUFACTURING IN SPACE:
THE NBS PROGRAM OF GROUND BASED RESEARCH

By

E. Passaglia, R. L. Parker
National Bureau of Standards
Washington, D. C. 20234

ABSTRACT

NBS work for NASA in support of NASA's Space Processing Program will be described. The objectives of the NBS program are to perform ground-based studies of those aspects of space that could possibly provide a unique environment for making materials more perfect or more pure. The approach taken deals primarily with experimental and theoretical studies of the possible effects of the absence of gravitational forces on those materials preparation processes where the presence of gravity may be important in reducing perfection or purity. The materials preparation processes studies comprise six tasks in the areas of crystal growth, purification and chemical processing, and the preparation of composites. They are:

- (1) Crystal Perfection in Czochralski Growth
- (2) Evaporative Purification of Ultrahigh Purity Materials
- (3) Vacuum Effects in the Preparation of Composite Materials
- (4) Melt Shape in Weightless Crystal Growth
- (5) Vapor Transport Synthesis and Crystal Growth of Oxides
- (6) Surface Traction and Other Surface Phenomena

STUDIES OF LIQUID FLOATING ZONES IN ZERO
GRAVITY ON SL-IV, THE THIRD SKYLAB MISSION

By

J. R. Carruthers
Bell Laboratories
Murray Hill, New Jersey 07974

ABSTRACT

Cylindrical liquid floating zones were suspended between two axially aligned circular discs by astronaut, Dr. E. G. Gibson, on the third Skylab mission. The surface stabilities of zones of various lengths were studied for both static and rotation conditions and for additional solid access to the zones. In addition liquid flow patterns were observed under the transient spin-up as well as steady-state rotation conditions. Information was obtained on the vibration response of the zones and the influence of surface tension and its gradients on the behavior of the zones in zero gravity. These studies will augment work already performed under simulated zero-gravity conditions to give some insight into flow pattern problems which cause undesirable radial solute segregation distributions in floating zone crystal growth. In addition, the basic information has been acquired to develop this technique for future materials space processing applications on the Space Shuttle.

LOW-GRAVITY PARTICLE DISPERSION IN LIQUID METALS

By

J. Raat, S. Kaye
General Dynamics
Convair Aerospace Division
San Diego, California 92138

ABSTRACT

A new technique is proposed for fiber immersion in liquid metals at low gravity, utilizing low pressures and elevated temperatures to induce fiber-matrix bonding.

A method for correlating gravity-induced particle segregation rates is presented; a simple segregation law for slender fibers is checked against experimental results.

A criterion is derived for estimating the importance of particle centrifugal drifting.

OXIDE GLASS PROCESSING IN SPACE

By

R. A. Happe
Space Division, Rockwell International Corporation
Seal Beach, California 90740

And

L. E. Topol
Science Center, Rockwell International Corporation
Thousand Oaks, California 91360

ABSTRACT

Processing in space theoretically permits containerless melting of refractory materials and cooling to spherical-shaped boules without the need for a solid mold. The absence of a melting container or mold, sources of contamination as well as heterogeneous nucleation sites, should make possible the production of glasses of compositions normally available only in the solid (crystalline) state. Such glasses should have properties which permit the design of improved lenses for advanced optical systems. The possibilities for utilizing containerless melting and cooling to produce unusual glasses have been explored for several years.

The highlights of the experimental effort are presented beginning with the preparation of 100 to 800 micron diameter spherules of glass of unusual oxide compositions by a laser spin-melting and free-fall cooling technique. The indices of refraction and Abbe numbers of most of the glasses produced were measured.

More recent experimental work is employing air suspension of boules which are melted by a focussed CO₂ laser beam and cooled in air suspension without contact with crystalline material during any part of the preparation cycle. Such a technique shows promise of extending the size of experimental boules to 1/4 inch diameter or larger.

Experimental work performed to date appears to be confirming the validity of the original premises.

SPACE PROCESSING OF CHALCOGENIDE GLASSES

By

D. C. Larsen, M. A. Ali, W. B. Crandall
IIT Research Institute
Chicago, Illinois 60616

ABSTRACT

The weightless, containerless nature of space manufacture has the potential for eliminating causes of optical non-homogeneity and contamination in chalcogenide glass systems. Manufacture in space should thus eliminate (1) melt disproportionation present in 1-G Earth manufacture, (2) contamination by oxygen and other elements deleterious to infrared transmission and (3) heterogeneous nucleation due to the presence of a crucible during melting. A program is described to demonstrate the feasibility of space manufacture of improved chalcogenide systems. Optimum methods, techniques and equipment are being defined through evaluation of amorphous character, purity, and homogeneity parameters at various stages of the glass forming process (i.e. from the raw material stage through the melt-quenching stage). Various terrestrial experiments are being conducted that will predict the improved levels of homogeneity, purity, and amorphuousness attainable in space. Initial experiments are being conducted with the As_2S_3 system. Results will be extended to more complex systems such as Si-As-Te-Sb.

EUTECTIC SOLIDIFICATION IN SPACE

By

F. C. Douglas, F. S. Galasso
United Aircraft Research Laboratories
East Hartford, Connecticut 06108

ABSTRACT

Unidirectional solidification of eutectic systems provides aligned composite structures with possible applications in electronic, magnetic, optical, and structural fields. The major emphasis in the development of DSC (directionally solidified composite) materials has been for structural purposes where the gross properties are relatively independent of microstructural perfection. Attention is now being given to methods of solidification which result in fewer microstructural defects in order to provide usable materials for non-structural applications. In addition, it is desirable to be able to unidirectionally solidify complex structures such as turbine blades while maintaining high quality microstructure.

The opportunity to prepare DSC materials in the low-gravity environment of space has stimulated research on the advantages such processing could yield. In particular, elimination of density-driven convection and reductions in the mass of containers due to near-zero gravity could contribute significantly to the processing of off-eutectic compositions and of large parts, respectively. Further, it is felt that superior thermal control could be obtained when the mass of the container can be made negligible with respect to the material being processed. Reductions in the microstructural defects will be the result of improved process control.

This paper reviews the investigations conducted for the processing of eutectic systems in space. Much of the work discussed was conducted under the sponsorship of Marshall Space Flight Center.

CONCENTRATION CHANGES DURING EUTECTIC SOLIDIFICATION

By

B. N. Bhat
National Research Council Research Associate
Process Engineering Laboratory
Marshall Space Flight Center
Alabama 35812

ABSTRACT

When the liquid aluminum-copper eutectic alloy is held in a temperature gradient, copper is observed to migrate to the colder regions. This is known as thermotransport, thermal diffusion or Soret effect. The flux of copper atoms due to thermotransport is determined for the eutectic solidification experiment of the Skylab and its influence on the composition of the solidified alloy is calculated. It is found that thermotransport causes precipitation of copper-rich Θ phase at the solid-liquid interface. The amount of Θ phase formed is found to be a function of the rate of solidification. Thermotransport is significant at slow rates of growth (in the range of 10^{-6} - 10^{-5} cm/sec) but it is negligible at higher rates of solidification (greater than 10^{-4} cm/sec). These findings are supported by the results of the Skylab experiments. There is a good agreement between the calculated and observed variation in composition.

A method is suggested whereby the concentration changes due to thermotransport may be corrected. This is called the modified Bridgman method. According to this method the eutectic alloy is subjected to a known temperature gradient in the molten condition for a known period of time. Controlled amounts of segregation can be introduced by this process. The sample is then solidified. It is then inverted and grown in the usual manner. The concentration changes occurring during growth are now compensated for and hence a more homogeneous alloy is obtained.

EFFECT OF GRAVITY ON LIQUID PHASE SINTERING

By

T. Mookherji, W. B. McAnelly
Teledyne Brown Engineering
Huntsville, Alabama 35807

and

E. C. McKannan
Astronautics Laboratory
Marshall Space Flight Center
Alabama 35812

ABSTRACT

The objective of the present work is to study the utilization of the unique conditions offered by the earth orbital environment in material processing involving both solid and liquid phases -- such as liquid phase sintering.

To achieve this, an experiment development program involving both test and theoretical work was initiated. Experimental work using material combinations selected such that maximum information about the effect of gravity can be derived has been conducted. Wetting of the solid phase by the liquid during sintering is an important phenomena in liquid phase sintering and gravity has influence on both capillary phenomena and density segregation; hence, material combinations were selected such that these two effects can be suitably studied. The experimental work is meant to form the basis for similar comparative work done under low-g conditions.

The results of electrical and metallographic measurements and their correlation with a theoretical model is presented here.

IMMISCIBLE MATERIALS AND ALLOYS

By

A. J. Markworth, S. H. Gelles, J. J. Duga
Battelle
Columbus Laboratories
Columbus, Ohio 43201

and

W. Oldfield
Materials Research and Computer Simulation
Westerville, Ohio 43081

ABSTRACT

Processing multiphase materials in space is discussed in terms of potential benefits in resulting properties or combinations of properties. Such processing may produce unique superconductors, bearing alloys, catalysts, superplastic materials or dispersion-strengthened metals.

Three basic techniques for producing multiphase systems which are amenable to space processing are described. One involves the mixing of inert particles, whiskers or rods in a liquid. The other two deal with the decomposition of a liquid by precipitation or by eutectic, monotectic or peritectic reactions.

Efforts at Battelle's Columbus Laboratories are discussed which have been concentrated on the precipitation of liquid droplets in a host liquid during cooling through a miscibility gap. The major objective is to study the agglomeration of the droplets both experimentally and through computer simulation in order to understand the mechanisms involved and to deduce the effect of gravity on the droplet distribution and resulting structure of the solidified material.

THE ELECTRICAL PROPERTIES OF ZERO-G PROCESSED MULTICOMPONENT MATERIALS

By

G. H. Otto
Physics Department
University of Alabama in Huntsville
Huntsville, Alabama 35807

and

L. L. Lacy
Space Sciences Laboratory
Marshall Space Flight Center
Alabama 35812

ABSTRACT

When dispersed or mixed immiscibles are solidified on earth, a large amount of separation of the constituents takes place due to differences in densities. However, when the immiscibles are dispersed and solidified in zero-gravity, density separation does not occur, and unique composite solids can be formed with many new and promising electrical properties. By measuring the electrical resistivity and superconducting critical temperature T_c of zero-g processed Ga-Bi samples, it has been found that the electrical properties of such materials are entirely different from the basic constituents and the ground control samples. Our results indicate that space processed immiscible materials may form an entirely new class of electronic materials.

This paper will also briefly review the interesting electrical properties of directionally solidified Al-Cu eutectic, a two component alloy consisting of Al and Al_2Cu which was processed on Skylab (M566).

PROJECTED FUTURE SPACE PROCESSING ACTIVITIES

By

James H. Bredt
Office of Applications
National Aeronautics and Space Administration
Washington, D. C. 20546

ABSTRACT

Through its early years the NASA space processing program has been devoted primarily to exploring the potential for useful space applications in materials science and technology and developing ways to pursue applications whose potential seems to be high. This work has identified several technical areas in which space processing can make substantial contributions in two types of application: applied research and manufacturing operations. The Skylab experiments have strikingly verified the utility of space for research purposes; their high proportion of unexpected results indicates that space will be a fertile field for new discovery, and the ASTP experiments are expected to confirm this finding.

We can therefore expect workers in applied research to show a high degree of interest in future flight opportunities, and the program's main assignment in this area will be to support that interest with adequate experiment capabilities. We plan to do this by conducting an interim sounding rocket program to prepare both NASA and the user community for the large increase of activity that the Shuttle/Spacelab system will bring, and by involving users actively in planning our equipment inventory for the early years of Shuttle operations.

Useful manufacturing applications are farther in the future, and progress in this area has been correspondingly slower. A high proportion of space processing experiment results will be applicable to space manufacturing, so that increased experiment activity will automatically contribute to manufacturing goals. However, we must expect industrial interest to center in the short run on research results that can be applied to current needs on the ground. As private activity develops, the NASA program will need to shift increasingly from general space utilization to activities specifically directed toward long-range manufacturing prospects.